

CAPACITIVE VACUUM MEASURING CELL

Background of the Invention

The present invention generally relates to a capacitive vacuum measuring cell.

It is known that pressures or pressure differences can be measured by applying pressure to a thin membrane and measuring its deflection. A known and suitable method for measuring the deflection is to design the membrane arrangement as a variable electrical capacitance where the capacitance change which correlates with the pressure change is evaluated by measurement electronics in the known manner. The capacitance is created by arranging a thin, flexible membrane very close to another surface and by depositing an electrically conductive film on both mutually opposed surfaces or by fabricating them from electrically conductive material. When pressure is applied to the membrane, the deflection changes the distance between the two electrodes which leads to an analyzable capacitance change of the arrangement. Sensors of this type are mass-produced from silicon. The flat basic body, as well as the membrane, often consist entirely of silicon. There are also versions that are made of composite materials such as silicon with a glass substrate. Such sensors can be produced very economically. However, in vacuum applications, pressure sensors of this type are normally usable only for higher pressures in the range of approx. 10^{-1} mbar to several bar. High resolution at pressures below 10^{-1} mbar is no longer achievable with silicon. One of the reasons for this is that the silicon surface reacts with the environment, which impairs the sensitive sensor characteristic. Already water vapor that forms part of normal atmospheric air leads to a corresponding reaction on the surfaces. The problem becomes even more serious when the sensor is used in chemically aggressive atmospheres. For this reason, attempts were made to protect such silicon sensors against external influences by passivating the surfaces. Attempts were also made to deposit protective coatings on the surfaces in order to improve the durability and the resistance against chemically aggressive environments as described in DE 41 36 987. Such measures are costly and, in the case of mechanically deformable parts such as membranes, have only limited success, in particular in highly aggressive media such as fluorine, bromic acid and their compounds which are typically used in vacuum etching processes.

1 For this reason, attempts were made to build vacuum measuring cells entirely from
2 corrosion resistant materials such as Al_2O_3 . A known arrangement of this type is shown in Fig. 1.
3 The vacuum measuring cell consists of a ceramic plate (20) above which a membrane (22) is
4 arranged with a small gap between the two of them and a fusible seal (21) between the ceramic
5 plate (20) and the edge of the membrane. The ceramic plate (20) together with the membrane
6 (22) forms a reference vacuum chamber (25) that is evacuated down during the manufacturing
7 process through a pumping port and which is sealed with a seal (28). The mutually opposed
8 surfaces of the ceramic plate (20) and the membrane (22) inside the reference vacuum chamber
9 (25) are coated with electrically conductive material and connected to insulated external terminals
10 on which the capacitance signal can be evaluated by means of an electronic device (not shown in
11 the illustration). To achieve corrosion resistance, plate (20) and membrane (22) are both made of
12 ceramic material such as Al_2O_3 . This vacuum measuring cell in turn is arranged in a vacuum-tight
13 housing (23) that features a port (24) which is connected to the media to be measured. Via port
14 (24) of the vacuum measuring cell, the resulting measurement vacuum chamber (26) is sealed off
15 against the membrane (22) by means of an elastomer seal (27) so that the pressures to be
16 measured reach only the surface of the membrane (22). For the purpose of sealing, the entire cell
17 is pressed via the ceramic plate (20) and membrane (22) against the elastomer seal (27). Up to
18 now, vacuum measuring cells of this type have been usable only for higher pressures in the range
19 of 0.1 mbar to 100 bar. In addition, this design leads to stress in the materials which, at lower
20 pressures, for example < 1 mbar, significantly impairs the reproducibility of measurement results
21 and the resolution. The ceramic membranes (22) used so far have a thickness ranging from 279
22 μm to 2540 μm . Such designs are not suitable for achieving wide measurement ranges, in
23 particular low pressures of 0.1 mbar to 10^{-6} mbar. In addition, designs of this type, as disclosed
24 also in U.S. Pat. No. 5,553,502, are costly.

25 The objective of the present invention is to eliminate the disadvantage of the current state
26 of the art. In particular the objective of the present invention is to implement an easy-to-produce,
27 economical vacuum measuring cell that is suitable for measuring pressures from 10^{-6} mbar to 1000
28 mbar, in particular from 10^{-6} mbar to 1 mbar, with an accuracy of better than 1%, preferably

1 better than 0.3% of the measured value. The measurement range can be covered or subdivided
2 into several vacuum measuring cells or membrane versions according to the invention. In addition
3 this vacuum measuring cell shall be corrosion resistant to aggressive media, have a compact
4 design, and be economical to manufacture.

6 Summary of the Invention

7 With respect to the generic vacuum measuring cell, the problem is solved through the
8 characteristic features of patent claims 1 and 19. The dependent patent claims relate to preferable
9 other versions of the invention.

10 The capacitive vacuum measuring cell according to the invention is made entirely out of
11 ceramic, in particular Al_2O_3 . This results in high corrosion resistance and long term
12 reproducibility. Only in the areas where sealing is required or where feedthroughs are provided
13 are small amounts of materials other than Al_2O_3 used, if the Al_2O_3 is not fused without addition of
14 the foreign material. A vacuum measuring cell consists of a first plate-shaped housing body above
15 which a membrane, sealed along its edges, is arranged so that it encloses a reference vacuum
16 chamber. On the side pointing away from the reference vacuum chamber, there is a second
17 housing body, also sealed along its edges, so that a measurement vacuum chamber is formed
18 there. This measurement vacuum chamber features a port for connecting the medium to be
19 measured. The surface of the first housing body and the membrane that form the reference
20 vacuum chamber are coated with an electrically conductive film, for example, gold, and constitute
21 the electrodes of the capacitance measuring cell. The electrodes are led out, for example, through
22 the first housing body or through the sealing area in the edge zones. The essentially parallel
23 electrode surfaces are spaced apart from 2 μm to 50 μm . Sealing of the membrane in the edge
24 zone against the two housings is preferably achieved through welding, for example, laser welding.
25 Highly suitable, and simple to use, is also a glass soldering and/or brazing material that is
26 corrosion resistant. Another possibility of achieving a sealing bond is to connect the housing parts
27 diffusively, for example, in the green body state in which the objective is to completely avoid
28 material other than Al_2O_3 .

1 The measuring cell arrangement according to the invention essentially allows a symmetric
2 design that avoids all stress in the housing. This is particularly important in order to achieve high
3 measurement sensitivity combined with high accuracy and reproducibility. It also allows the
4 utilization of a very thin ceramic membrane which is essential for reliably measuring vacuum
5 pressures lower than 100 mbar, and in particular lower than 10 mbar, by means of capacitive, all-
6 ceramic measuring cells. For this purpose, membrane thicknesses of 10 μm to 250 μm are
7 needed, where membrane thicknesses of 10 μm to 120 μm are preferred in order to achieve a very
8 good resolution. Typical membrane thicknesses are, for example:

- 9 • at 1000 Torr: membrane thickness 760 $\mu\text{m} \pm 10 \mu\text{m}$
- 10 • at 100 Torr: membrane thickness 345 $\mu\text{m} \pm 10 \mu\text{m}$
- 11 • at 10 Torr: membrane thickness 150 $\mu\text{m} \pm 10 \mu\text{m}$
- 12 • at 1 Torr: membrane thickness 100 $\mu\text{m} \pm 10 \mu\text{m}$
- 13 • at 0.1 Torr: membrane thickness 60 $\mu\text{m} \pm 10 \mu\text{m}$
- 14 • at 0.01 Torr: membrane thickness 40 $\mu\text{m} \pm 10 \mu\text{m}$

15 Such thin membranes are very difficult to manufacture and, after the sinter step, require at least
16 one additional smoothing step. It is also very important for the membrane to be sufficiently
17 helium tight which can be achieved only if the grain size of the membrane material is not too large
18 and remains within the range of $<20 \mu\text{m}$. Smaller grain sizes of $<10 \mu\text{m}$ are preferred, in
19 particular, those $<5 \mu\text{m}$. In all cases, the cross-section of the membrane viewed across the
20 thickness should contain at least two grains; the membranes are particularly tight if more than five
21 grains are on top of each other.

22 Another important criterion for achieving an accurate measuring cell is the planarity of the
23 membrane surface. The unevenness across the entire surface should in all cases not exceed 30%
24 of the electrode gap, preferably not more than 15%. This means that the unevenness across the
25 entire surface should not exceed 10 μm , preferably no more than 5 μm . The unevenness is defined
26 here as the difference between the lowest and the highest point. To achieve the desired long-time
27 stability, the purity of the aluminum oxide used for the membrane should be at least 94%, with
28 preferred values being above 99%.

1 To ensure that the quality of the membrane seal in the edge zone is not impaired, it is
2 advantageous to lead out the electrically conductive layers via feedthroughs that are arranged on
3 the first housing body, rather than directly through the membrane seal or fused joint.

4 To ensure accurate functioning of the measuring cell over a long period of time, the
5 reference vacuum chamber must have a high-quality vacuum with long-time stability. After
6 evacuation, a getter should be provided that is preferably arranged in a small volume on the first
7 housing and communicates with the reference vacuum chamber. This getter ensures that the
8 reference vacuum pressure is lower, preferably by at least one decade, than the pressure to be
9 measured. To prevent contaminations of the internal measuring cell space, a getter type that is
10 not evaporating should be chosen.

11 Measuring cells designed according to the invention can be very compact and economical
12 to produce. The diameter of such a cell can be between 5 and 80 mm where the measuring cell
13 preferably has a diameter of 5 to 40 mm. The thickness of such a cell is preferably in the range of
14 2 mm to 25 mm.

15 16 Brief Description of the Drawings

17 Fig. 1 is a prior art drawing of a known vacuum measuring cell built mainly from
18 corrosion resistant materials such as Al_2O_3 .

19 Fig. 2 shows a schematic cross-section of a capacitive vacuum measuring cell according to
20 the invention.

21 Fig. 3 shows an enlarged cross-sectional detail according to Fig. 2.

22 Fig. 4 shows a cross-sectional detail of a getter arrangement

23 Fig. 5 shows a cross-section of another getter arrangement version.

24 25 Detailed Description of the Preferred Embodiments

26 For manufacturing a functional measuring cell that possesses the aforementioned
27 characteristics, the specifications of the corresponding manufacturing process must be closely
28 followed. In particular, the manufacture of thin ceramic membranes requires special care. The

1 membrane as well as the complete unit should be entirely free of internal stress.

2 Suitable Al_2O_3 membranes are manufactured, as is usual in the ceramic industry, by first
3 mixing a slurry according to a specific recipe, and by thinly and evenly spreading the doughy mass
4 on a strip shaped carrier material, for example, a plastic foil. After drying, these layers are
5 inspected for irregularities such as bubbles or pits. This mass, which is not sintered yet, is referred
6 to as the green body. The desired membrane shape is cut out of the strip shaped green body
7 material, after which the material is still sticking to the plastic foil. For cutting, tools such as
8 knives are used, preferably a laser. Cutting or scoring of the green body requires particular care
9 that no dislocations or warping against the surfaces of the future ceramic membrane occur, as this
10 also influences the degree of surface unevenness. If a cutting knife is used, a pressing wheel can
11 be applied on the membrane side which prevents undue warping of the green body. Subsequently
12 the preferably circular cut membranes are separated from the foil strip by drawing off the latter,
13 for example, across an edge. The membranes are subsequently sintered in a furnace. For
14 sintering, the membranes are preferably placed on hard-sintered, flat Al_2O_3 plates that can be
15 stacked on top of each other, and sintered typically at 1630°C . The temperature is gradually
16 raised to 1630°C over a period of approx. 400 minutes, which corresponds to a temperature rise
17 of about 40°C per minute. The temperature is then held for a few minutes at this level, for
18 example, 6 minutes, and then slowly decreased at the rate of about 3°C per minute over 210
19 minutes to 1000°C , and in a second step, with a temperature reduction of about 6°C per minute
20 over about 170 minutes, to room temperature. The result is a ceramic membrane which, in
21 contrast to the green body, has a hard pure ceramic structure, and the additives of the green body
22 material have evaporated. After this sintering step, the membrane is very uneven and, at a
23 diameter of 40 mm, has a warpage of several millimeters.

24 In this condition, the membrane cannot be used due to the strong warpage and internal
25 stress in the material. The membrane must be smoothed in at least one additional step. For this
26 purpose, the membrane is again heated in the furnace. The membrane is carefully sandwiched
27 between massive and highly planar, hard-sintered Al_2O_3 plates (also "dead", that is, large-grained
28 Al_2O_3) which, for a membrane diameter of 40 mm, have a weight of several 10 to several 100

grams, or in the example about 60 grams, or are correspondingly weighted down. The temperature is slowly increased at 4°C per minute over 390 minutes to approximately 1570°C. After a short dwell time of several minutes, approximately 25 minutes at this temperature, the temperature is lowered slowly at approx. 5°C per minute over approx. 115 minutes until 1000°C are reached. Subsequently, the temperature is lowered at approx. 6°C per minute over about 166 minutes until ambient temperature is attained. After this smoothing step, the membrane has only a very small amount of residual warpage of a few tenths of a millimeter. Important in this smoothing step is that the temperature is not raised as high as in the first sintering process, preferably up to a temperature which is at most 100°C lower than the sintering temperature. To achieve excellent results required for the measuring cell to be built, this smoothing heating step must be performed at least twice. For reasons of economy, these smoothing steps should be performed in such a way that no more than two such steps are needed. Particularly good results are achieved when the membrane is carefully separated from the plate between heating steps and redeposited in a slightly offset position. Preferably, the membrane is even placed upside down. The utilization of a stack of several flat plates with membranes sandwiched in between is particularly economical. After these steps, membranes are now available that have selectable thicknesses in the range of 10 µm to 250 µm, preferably <120 µm. With the process described above, membrane planarities can be achieved that are better than 10 µm across the entire surface, preferably even better than 5 µm. The mean grain size in the membrane material is less than 20 µm, preferably less than 10 µm, and even less than 5 µm is achievable. In this way, the requirement that at least 2 grains, preferably at least five grains, exist across the thickness, can easily be achieved. In this way, helium tight membranes, as required for vacuum measuring cell applications, can be produced. The membrane is now ready to be used for building the measuring cell.

The membrane, as well as a flat surface of the first housing body made of Al_2O_3 , are now coated with an electrically conductive film for creating the electrodes. For example, a metallic paint, for example a paint containing gold, can be used which, for example, is brushed or sprayed, preferably printed on. Another method is to create the electrically conductive layer by means of

1 evaporation coating, preferably by sputter coating. To allow the deposition of an accurate and
2 defined film, it is advantageous if, for example, a gold layer that initially is deposited with a
3 relatively large thickness of about 1 μm , is subsequently thinned down in the inner area to approx.
4 5 nm by means of an etching process, preferably an ion etching or sputter etching process. In this
5 way, a thicker edge area is created which can compensate diffusion losses if, for example, a
6 soldering and/or brazing step is performed. A preferred process that is simple to handle in
7 practice is to first deposit a thin layer of several nm across the entire surface and subsequently a
8 thicker layer of gold at the edge by means of screen printing (that is, a combination process and
9 different film thicknesses). Membranes or housings processed in such a way are subsequently
10 tempered at temperatures of several 100°C, preferably in the range of 650°C.

11 The second ceramic housing which is arranged on the measurement side consists of a flat
12 ceramic plate which, on the membrane side, can have a flat recess in order to form a sufficiently
13 large vacuum chamber. The connection port is connected to this ceramic housing by means of
14 soldering and/or brazing, bonding or gluing, preferably by means of glass soldering and/or
15 brazing, in such a way that the connection opening can communicate with the future measurement
16 vacuum chamber.

17 In the peripheral area where the seal is created, the membrane is coated on both sides with
18 a glass paste, preferably by means of screen printing. After drying, the membrane with the glass
19 paste is baked in an oven at several 100°C, preferably at about 670°C. Subsequently, the glass
20 surface is polished on both sides and, thereby preferably also, the future electrode spacing is
21 defined.

22 With the aid of said coating process, the upper ceramic housing on the electrode side can,
23 on the external surface, additionally be coated with an electrically conductive film in order to
24 achieve shielding. Also here, the connection points are formed on the housing. In an additional
25 step, the drill holes for the electrical feedthrough of the electrode connections are metallized,
26 preferably with silver.

27 In a test phase, the first housing with the electrode and the feedthroughs, together with the
28 deposited membrane, is checked for tightness and for correct electrode distance. Subsequently,

1 the lower housing part is mounted, and the entire assembly is loaded with weight in order to test
2 the function and distances. In a mounting frame, the getter connection may additionally be
3 mounted, and under a load weight of about 200 grams, the glass seals are baked at several 100°C,
4 preferably about 630°C. Subsequently, a test is performed to check that the required distances
5 are maintained. If necessary, the membrane spacing can be corrected through additional weight
6 loading or relieving, and an additional firing process. The sealing process must be executed very
7 carefully and, as mentioned, no stress should occur in the measuring cell arrangement.
8 Alternatively also, direct bonding can be used in place of glass or other sealants, preferably laser
9 bonding.

10 The various features of novelty which characterize the invention are pointed out with
11 particularity in the claims annexed to and forming a part of this disclosure, and are entirely based
12 on the Swiss priority application no. 1997 2950/97 filed December 23, 1997.

13 The invention is described schematically based on the following illustrations which serve
14 as examples:

15 Fig. 2 shows a schematic cross-section of a capacitive vacuum measuring cell according to
16 the invention.

17 Fig. 3 shows an enlarged cross-sectional detail according to Fig. 2.

18 Fig. 4 shows a cross-sectional detail of a getter arrangement.

19 Fig. 5 shows a cross-section of another getter arrangement version.

20 A capacitive measuring cell made of Al_2O_3 according to the invention with a structure essentially
21 symmetrical about the membrane is illustrated by the cross-section in Fig. 2. The first housing (1)
22 consists of a ceramic plate made of Al_2O_3 which along its edges is tightly bonded at a distance of
23 2 μm to 50 μm relative to the ceramic membrane (2) and which encloses a reference vacuum (25).
24 The distance between the two surfaces is usually established directly during the assembly by
25 means of the sealing material (3) located between the membrane edge and the housing. In this
26 way, a completely plane housing plate (1) can be used. In the same way, a measurement vacuum
27 chamber (26) is formed in a second housing (4) on the opposite membrane side; this vacuum
28 chamber is accessible for the media to be measured via a connecting port (5) through an opening

1 in the housing (4).

2 Fig. 3 shows an enlarged cross-sectional detail of the edge zone of a measuring cell. The
3 seal (3) on both sides of the membrane (2) defines, as mentioned above, the distance of the two
4 housings (1 and 4). This seal consists, for example and preferably, of glass paste that is easy to
5 handle and can, for example, be applied by means of screen printing. In a typical measuring cell
6 with an external diameter of 38 mm and a free internal membrane diameter of 30 mm, the distance
7 (3) is approx. 2 to 50 μm , preferably 12 to 35 μm . In this example, the first housing (1) has a
8 thickness of 5 mm, and the second housing (4) a thickness of 3 mm. The inner area of the second
9 housing (4) is preferably designed with an approx. 0.5 mm deep recess, as shown in Fig. 2, in
10 order to enlarge the measurement vacuum chamber (26). On the reference vacuum side, the
11 membrane (2) and the housing (1) are each coated with an electrically conductive film (7). These
12 two films are not electrically interconnected. Films (7) can, for example, be painted on, printed
13 on, sprayed on, or be deposited by means of a vacuum process.

14 Another version of a getter arrangement is illustrated in Fig. 5 where a contacting agent
15 (12), preferably a soldering and/or brazing material, is placed between getter (10) and cover (8) in
16 order to achieve better thermal contact. Excellent thermal conductivity is required here so that
17 the getter (10) can be thermally activated via the cover (8) from the outside. In addition this
18 getter arrangement allows activation of the getter and subsequent soldering and/or brazing of the
19 cover (8) in a single work step, provided the activation temperature of getter (10) and the
20 soldering and/or brazing temperature of the soldering and/or brazing material (9) are essentially
21 the same.